

Advances in Non-contact Measurement of Creep Properties

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Abstract:

Our team has developed a novel approach to measuring creep at extremely high temperatures using electrostatic levitation (ESL). This method has been demonstrated on niobium up to 2300°C, while ESL has melted tungsten (3400°C).

High-precision machined spheres of the sample are levitated in the NASA MSFC ESL, a national user facility, and heated with a laser. The laser is aligned off-center so that the absorbed photons transfer their momentum to the sample, causing it to rotate at up to 250,000+ RPM. The rapid rotation loads the sample through centripetal acceleration, causing it to deform. The deformation of the sample is captured on high-speed video, which is analyzed by machine-vision software from the University of Massachusetts. The deformations are compared to finite element models to determine the constitutive constants in the creep relation. Furthermore, the non-contact method exploits stress gradients within the sample to determine the stress exponent in a single test. This method was validated in collaboration with the University of Tennessee for niobium at 1985 °C, with agreement within the uncertainty of the conventional measurements.

A similar method is being employed on Ultra-High-Temperature ZrB₂- SiC composites, which may see application in rocket nozzles and sharp leading edges for hypersonic vehicles.

Advances in Non-contact Measurement of Creep Properties



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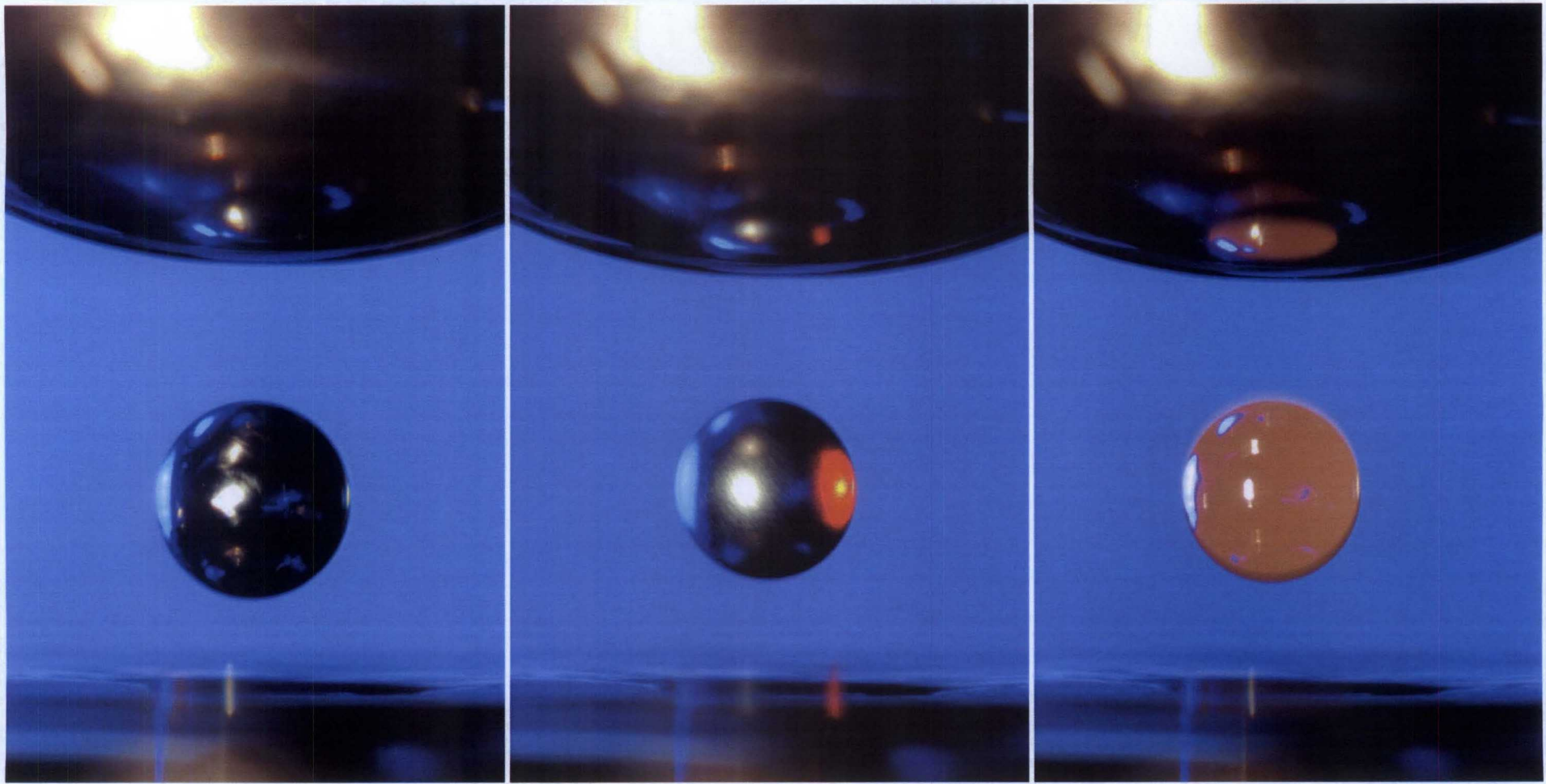
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ESL Team

- MSFC Science Team:
 - Trudy Allen, ICRC
 - Glenn Fountain, Qualis
 - Dr. Jan Rogers, EM50
 - Bill Witherow, EV43
- External Collaborators:
 - Dr. Robert Hyers, University of Massachusetts, Amherst
 - Dr. Peter Liaw, University of Tennessee Knoxville



ESL Team Members



- ESL provides an ideal method for study of high-temperature materials.
- Levitated samples do not contact a container and will not be contaminated by the container or react with it. Only the sample is heated, not the instrument and instrumentation.
- Pyrometry, or non-contact temperature measurements, employed in ESL can access higher temperatures than possible with thermocouples.
- Method provides a means to study materials at temperatures of interest to advanced propulsion (ESL can provide data over 3400 °C) that conventional methods cannot.

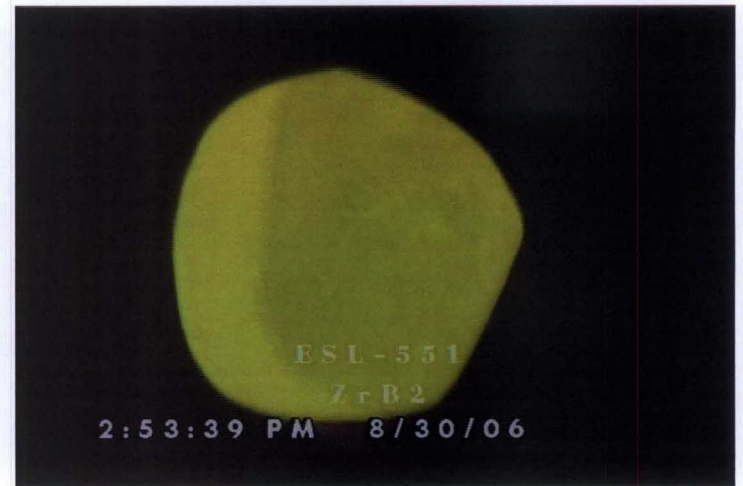
Materials Processed via ESL



Glass Melt
with
Crystallites



Metals and
Alloys

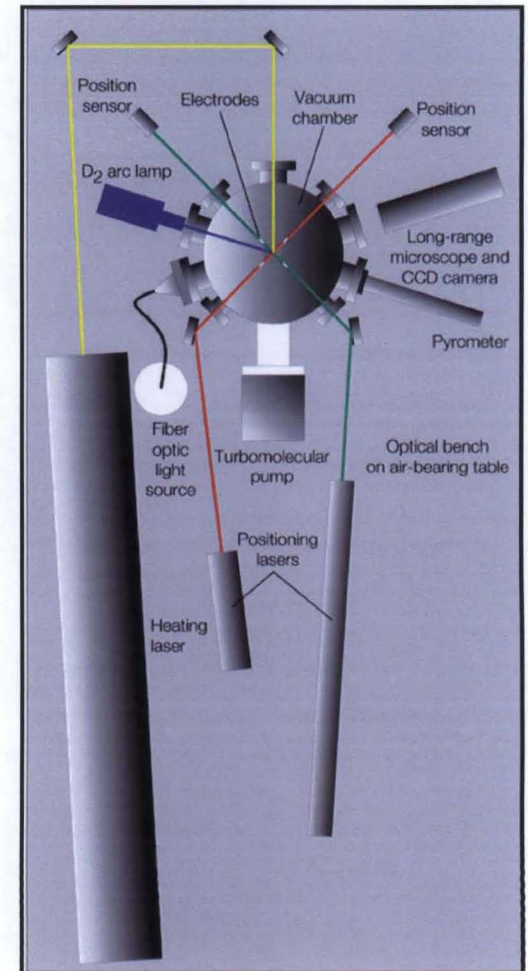


Ceramics, including
pressed materials
like ZrB_2

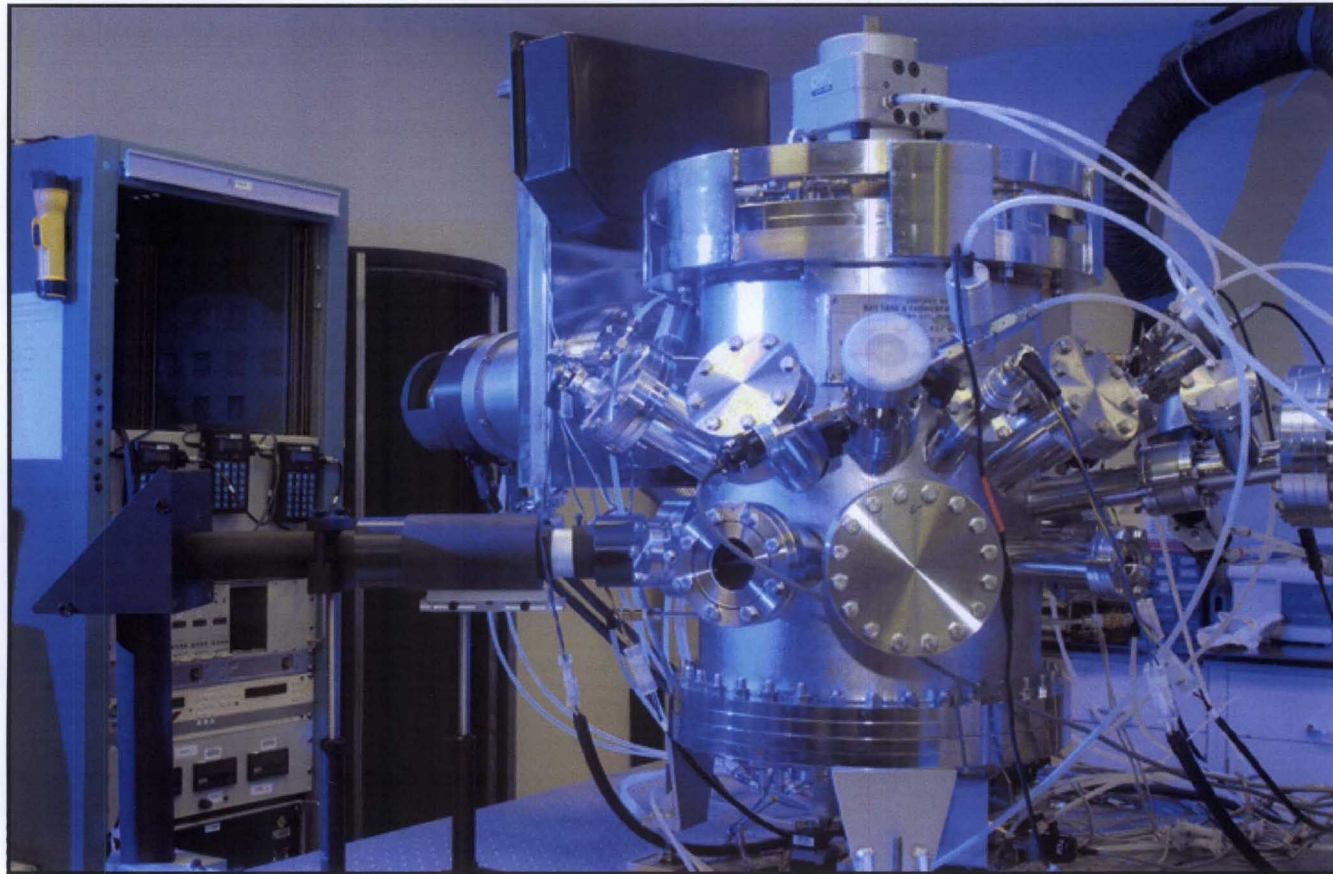
Other materials include polymers, semiconductors, solids, melts, and liquids.

MSFC ESL Facility

- The MSFC ESL Facility is a materials characterization facility that provides materials characterization data to users.
- Data files for thousands of melt cycles and hundreds of samples have been delivered to investigators, resulting in the development of new alloys, glasses, and numerous technical papers and journal articles.
- The MSFC ESL facility can provide measurements of thermophysical properties, which include creep strength, density and thermal expansion, emissivity, specific heat, and phase diagrams. For melts, viscosity and surface tension can be measured.
- Data can be obtained at ultra-high temperatures for materials being developed for propulsion applications.
- Samples: 2-3 mm diameter spheres (30-70 mg)
- Heated by lasers: 200W Nd:YAG or 300W CO₂



ESL System for Operations UHV - 5 atm



Non-contact Temperature Measurement Tools

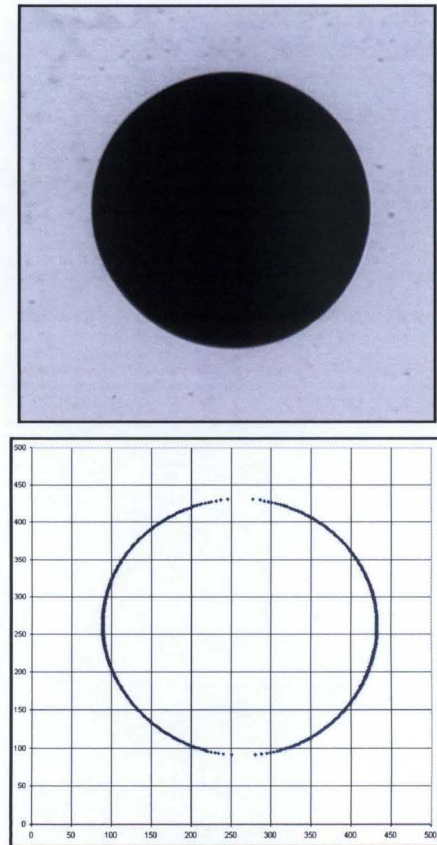
- Single wavelength/band pyrometers for the range: 200-3500 °C
- Two wavelength pyrometers for the range: 700-1400 °C
- Polarimeter to provide spectral emissivity at 670 nm
- Blackbody calibration source with operating range: 600-3000 °C
- Multi-wavelength spectropyrometers from FAR Associates provide temperature measurements with no operator input, even when the target's surface characteristics change with temperature or processing for the ranges:
 - 800-4000 °C
 - 300-2000 °C.

ESL Facility Capabilities

- Phantom V7 provides 12-bit monochromatic images with 800x600 pixel resolution at rates up to 160,000 frames per second.
- Redlake Motion ProModel 10,000 provides 8-bit monochromatic images with 1280x1024 pixel resolution at rates up to 10,000 frames per second.
- Optical viewports are available on the chambers for user-provided equipment.
- Sample load lock and carousel mechanism support high-throughput processing.
- Processing at 10^{-8} Torr or at pressures up to 5 atm
- An arc melter is available for sample fabrication.
- Additional capabilities: RGA and mass scales with 1-microgram resolution available for pre- and post-process weighing to determine mass losses.

Density/Creep Data Analysis

- Machine vision with subpixel interpolation
- Computer detects edges, calculates volume vs. time with 9-parameter fit
 - Needs good contrast
- Correlated with pyrometry, known mass gives density vs. temperature
- Typical precision: ~ 0.1% liquid, ~0.05% machined sphere, ~2% sample solidified in ESL
- Standard ESL backlighting uses a white light source.
- At high temperatures, sample becomes a very bright, radiant object and contrast becomes poor.
- Illumination system designed for high-temperature studies using blue laser provide good edge detection to 2800 °C.



Backlit sample showing excellent contrast and computer detected edge

R.C. Bradshaw, R.W. Hyers, *et al.*, UMass

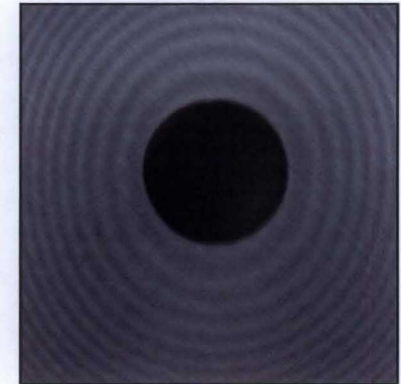
Imaging Ultra-High Temperature Samples



W ~2450 °C, bright,
radiant sample



W ~2800 °C, neutral
density filter added to
improve image



W ~**2800 °C**, **backlit with
blue laser, new limit for
edge detection**, higher
temperature possible with
laser upgrades

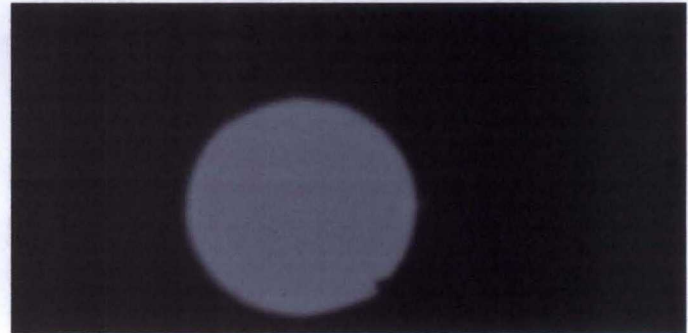
Color images from observation camera

B&W images for thermophysical property measurements from high-resolution,
high-speed camera

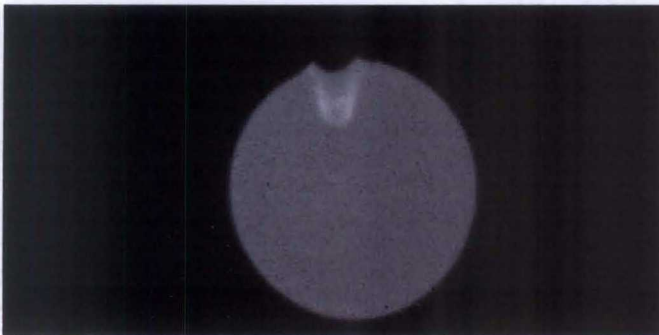
Sample Fabrication Methods and Shapes



Machined by general-purpose machine shop from Nb rod



Arc melted Nb by ESL personnel



Nb ESL melted and scribed



Nb machined by Industrial Tectonics Inc., precision sphere manufacturing specialists

Motivation for High-Temperature Creep Studies

- ❶ **Creep** is the deformation of a material resulting from prolonged stress.
- ❷ Creep resistance at high temperature is an important property for many advanced materials applications, such as propulsion systems design.
- ❸ Creep can induce component and system failures because of:
 - **Shape change:** creep caused failures in hot stage turbines of aircraft.
 - **Rupture:** creep caused failures in steam turbines for power generation.
- ❹ Creep/deformation processes are thermally activated:
 - Movement of dislocations easier at high temperature
 - Strain at high temperatures causes grain boundary migration and sliding/shearing at grain boundaries.
- ❺ Conventional methods for the measurement of creep resistance are limited to temperatures below **~1700 °C conventional lab or 1985 °C for Nb in high-temperature facility**. Advanced propulsion systems have targeted temperatures of ~3400 °C for operations. Rocket nozzles have to perform at temperatures that may exceed capabilities of standard test methods.

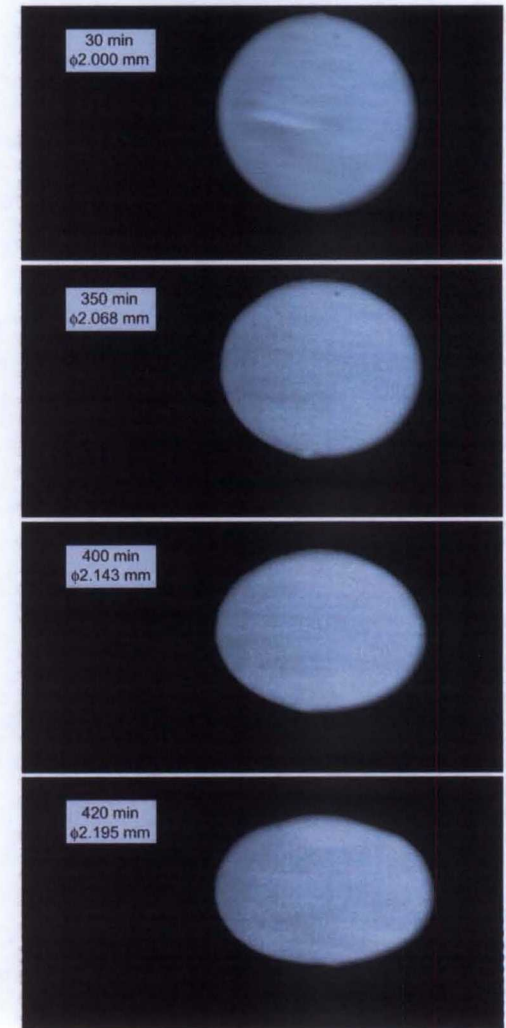
Non-Contact Creep Testing

- Creep tests performed via ESL
- Video shows deformation of Nb at 1985 °C
 - Maximum rotation rate:
~4,700 rotations per second
 - Compiled from clips of high-speed video at selected times during 435-minute experiment duration
- Photon pressure from heating laser induces rotation.
- Centrifugal acceleration causes creep deformation.
- Protocols, equipment, and analytical methods for creep developed and validated for testing at ultra-high temperatures



Ultra-High Temperature Creep Resistance via ESL

- Recent project completed to develop, validate, and utilize a new ESL technique for studies of creep resistance at ultra-high temperatures. Major project tasks included:
 - Use of centrifugal acceleration (from photon pressure) to induce creep in levitated samples
 - Capture of images of sample deformation at specified temperatures and times for analysis
 - Development of software for on-line rotation analysis and creep measurements
 - Development of predictive finite element model of stress/strain in samples
 - Validation tests using conventional ASTM test methods
 - Structural analysis of samples following ESL studies to examine deformation behavior and texture development.
 - Magnetic rotation apparatus under development
- Results from ESL tests show excellent agreement with results from validation testing at high-temperature materials facilities using ASTM Standard E-139.**
- Maximum temperature achieved for Nb creep using state-of-the-art high-temperature furnaces was 1985 °C
- ESL creep tests with Nb performed successfully at 2300 °C (Nb mp ~2486 °C); higher temperatures are possible.



Nb at 1985 °C

Non-contact Creep: Advantages and Limitations

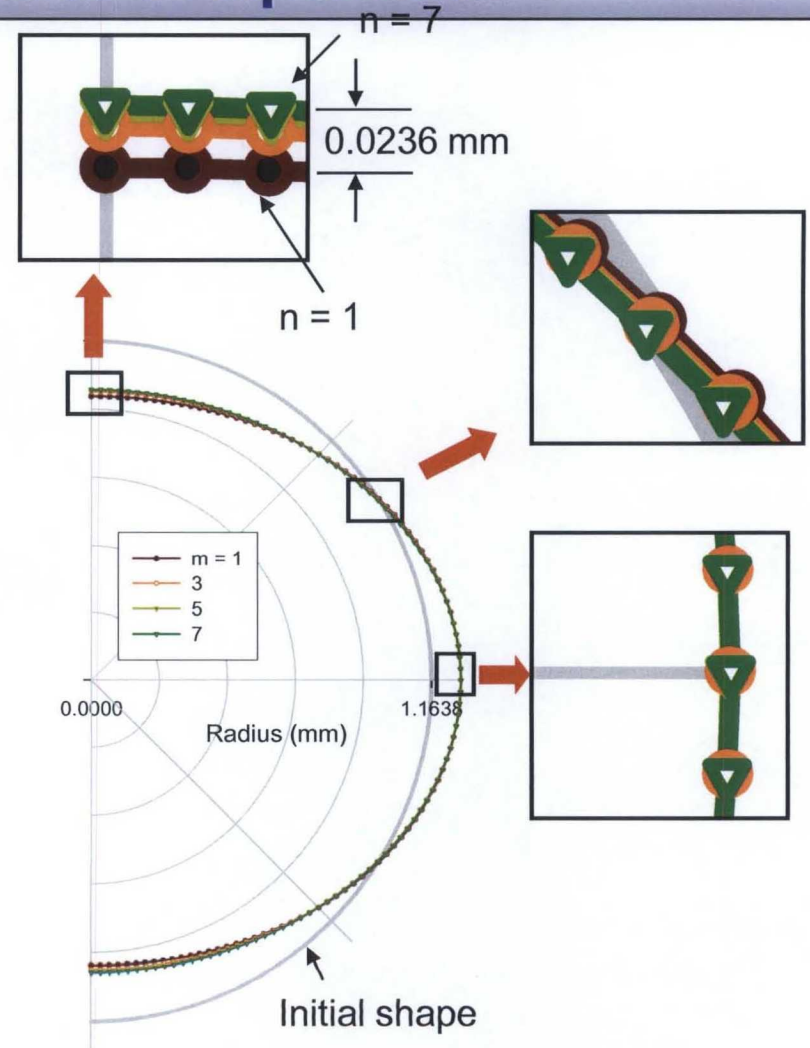
- Non-contact => No temperature limit
- Stress exponent from a single test
- Metals, Ceramics, Semiconductors
- 2-3 mm diameter samples
 - Very little material required, but
 - Microstructural length scale matters!

Parametric Study of Stress Exponent

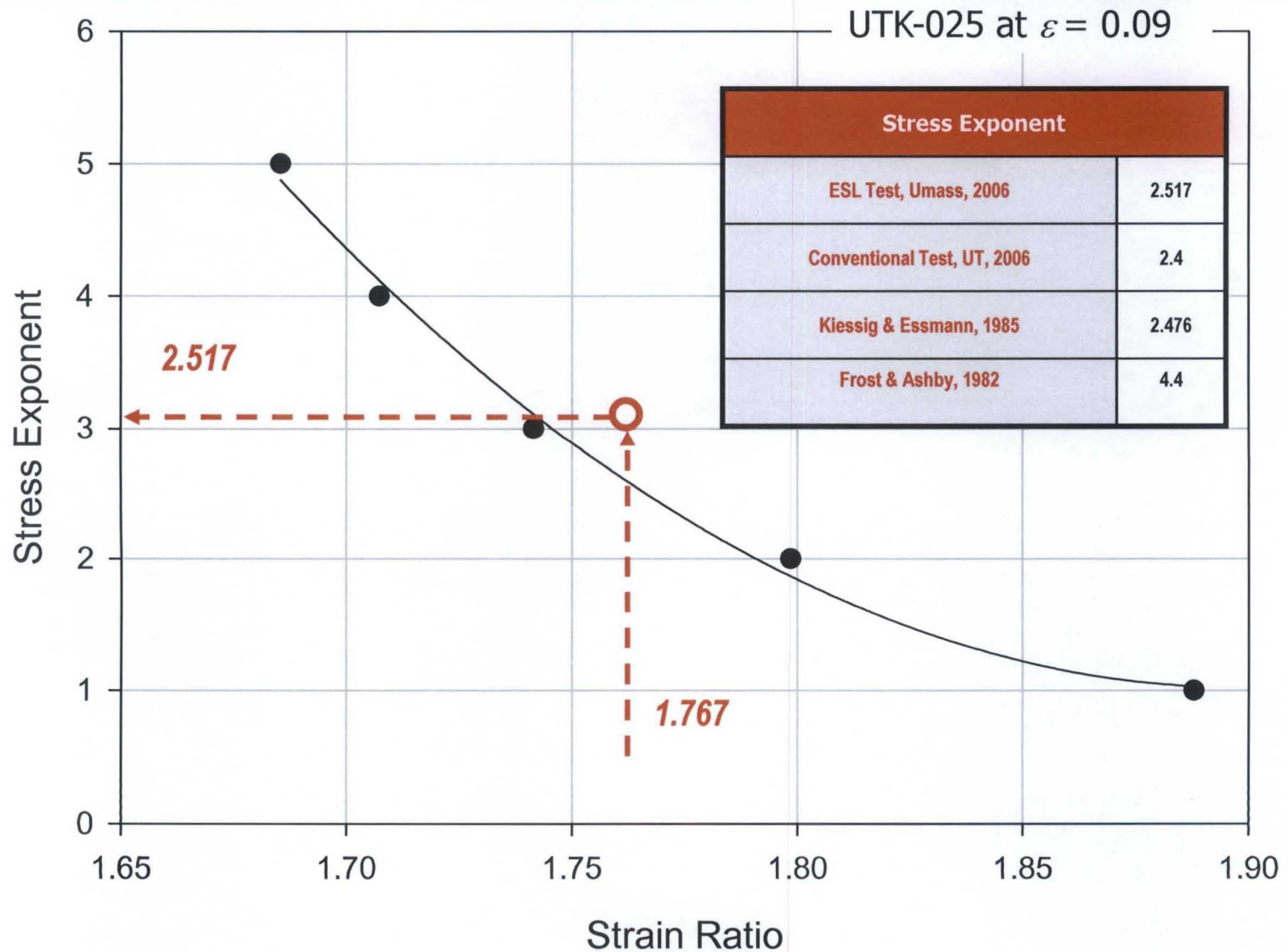
- Influence of the stress exponent on deformed shape investigated;
- FE models with different stress exponents run up to equatorial displacement of 0.1 mm ($e_{eq} = 0.086$);
- For the same equatorial strain, polar strain varies as a function of stress exponent;

$$\dot{\varepsilon} = C \sigma^n e^{\left(-\frac{Q}{RT}\right)}$$

- Intersection of the curves at around 45° measured from the axis of rotation to keep the area constant;
- The higher the stress exponent, the less polar strain;



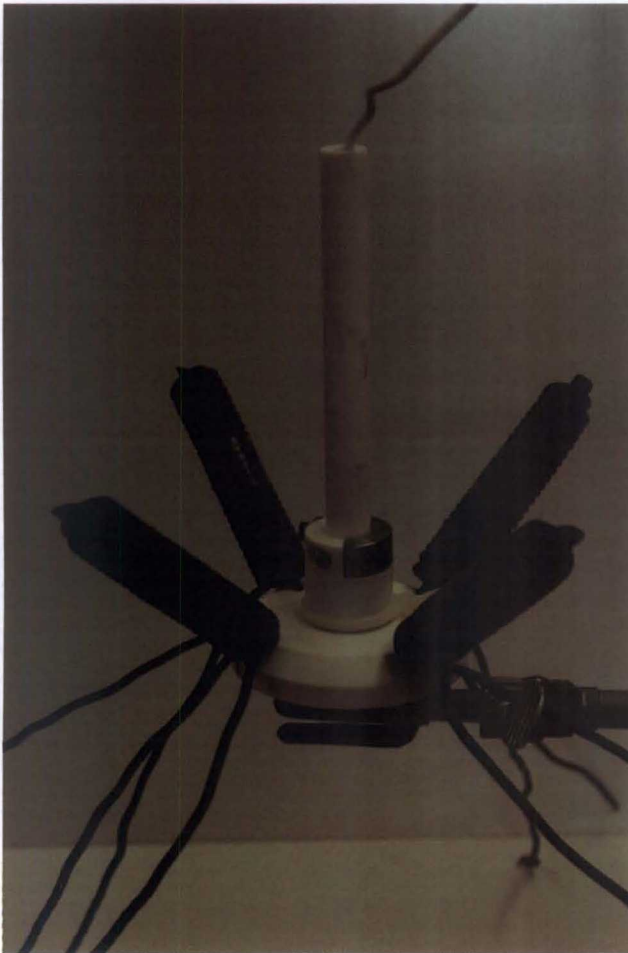
Determination of Stress Exponent



Rotation-induced creep

- Use of centrifugal acceleration to induce creep in levitated samples
- Currently using photon pressure from heating laser to induce rotation
 - Heating coupled with rotation
- Long times required to increase load (rotation rate) to induce deformation
- Preliminary studies with 40 mg sample of GE test sample did not rotate fast enough to show creep-deformation after 6 hours of testing using photon pressure
- Subsequent studies with smaller sample ~20 mg sample did not show deformation after six hours of processing.
- Magnetic rotation apparatus is being developed in order to increase acceleration rate.
 - Will permit independent control of rotation rate and sample temperature

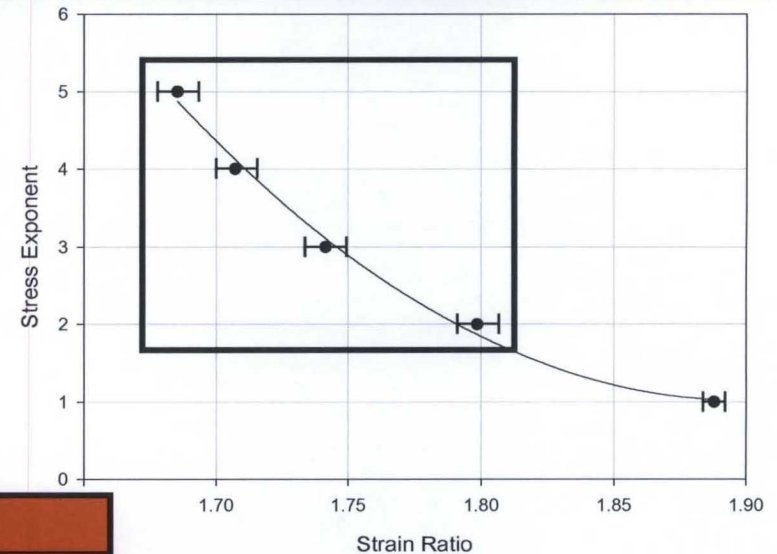
U Mass Magnetic Rotation Apparatus



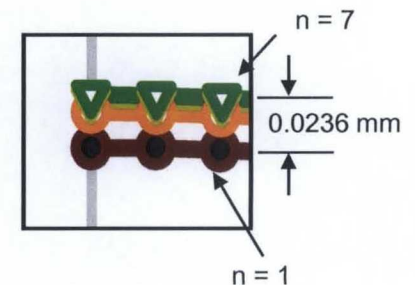
- Modeled the breadboard circuit.
- Measured a torque of 0.25×10^{-6} in-lb (2.8×10^{-8} N-m) with the breadboard model, which is over 600 times the torque produced by photon pressure at 2000°C .
- Prototype coils exceeded electrical performance of breadboard coils.
- Implementation of water cooling for prototype coils in progress for full benchtop performance test.
- Original amplifier failed during testing, replace with a higher-performance amplifier.
- Replacement amplifier failed and has been repaired by the factory.
- After repairs completed, amplifier has been performing to spec.

Accuracy of Stress Exponent

- Sensitivity analysis: measurement precision (170 PPM for edge position);
- The errors within ~1% for the most metals (stress exponent 2-5) at 0.09 strain;
- Accuracy increased by:
 - Using high-precision spheres;
 - Measurement at larger strains.



PPM	Error(%) Range	Stress Exponent				
		1	2	3	4	5
300	From	-0.18 %	-0.99 %	-0.89 %	-0.81 %	-0.75 %
	To	0.20 %	0.98 %	0.90 %	0.81 %	0.75 %
230	From	-0.14 %	-0.76 %	-0.70 %	-0.63 %	-0.58 %
	To	0.15 %	0.77 %	0.70 %	0.63 %	0.59 %
170	From	-0.10 %	-0.54 %	-0.50 %	-0.45 %	-0.42 %
	To	0.11 %	0.55 %	0.50 %	0.45 %	0.42 %
100	From	-0.06 %	-0.33 %	-0.30 %	-0.27 %	-0.25 %
	To	0.06 %	0.33 %	0.30 %	0.27 %	0.25 %
30	From	-0.02 %	-0.11 %	-0.10 %	-0.09 %	-0.08 %
	To	0.02 %	0.11	0.10 %	0.09 %	0.08 %



Results from ESL Creep Studies

- Creep deformation of Nb at 1985 °C was measured using both the ESL technique and a conventional testing method.
- The stress exponent from the ESL and conventional creep tests show good agreement with data from literature.
- The ESL method ***provides a unique capability for measuring creep at temperatures over 2000 °C, as required for numerous advanced aerospace applications.***
- ESL represents a promising technique for determining creep properties of ultra-high-temperature materials.

Method	Stress Exponent for Nb
ESL testing 2006	2.517
ASTM, furnace testing 2006	2.4
EML testing 1985	2.476
Extrapolation from low temp furnace data, 1982	4.4

Constitutive equation for creep in Nb, from Keissig, 1985:

$$\dot{\epsilon} = 2.64 \times 10^{-10} \sigma^{2.476} e^{-\left(\frac{55326}{T}\right)}$$

Ongoing Work for Creep

- New materials:
 - Nb superalloys, UHTC, refractory metals.
- Higher stresses:
 - Induction motor for sample rotation.
 - Enabling for measurements below $\sim 2000^{\circ}\text{C}$.
- Expanding collaborations.

Acknowledgements

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